IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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Applicant : Graetz et al.
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For : High-Capacity Nanostructured Silicon and Lithium Alloys Thereof
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MAIL STOP AMENDMENT
Commissioner for Patents
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DECLARATION OF JASON GRAETZ UNDER 37 CFR 1.132

Sir:

Jason Graetz hereby declares as follows:

1. I, Jason Graetz, am an inventor of the above-identified U.S. Patent Application No. 10/660,382.

2. I am presently a Research Scientist at Brookhaven National Laboratory.

3. I have experience and expertise in the fields of material science, spectroscopy and electrochemistry.

4. I have reviewed the Office Action of December 24, 2008 for U.S. Patent Application No. 10/660,382 and the references cited therein, including Abstract 257 of The 11th International Meeting on Lithium Batteries in Monterey, CA on June 23-28, 2002, entitled 'Li Insertion/Extraction Reaction of a Si Film Evaporated on a Ni Foil' (Takamura et al.); and Abstract 52 of

The 11th International Meeting on Lithium Batteries in Monterey, CA on June 23-28, 2002, entitled 'New active material structure in Si thin film electrodes for rechargeable lithium batteries' (Sayama et al.).

5. Figure 5A of U.S. Patent Application No. 10/660,382 provides electron energy loss (EELS) spectra showing the silicon $L_{2,3}$-edge of a standard silicon sample (Si), a standard SiO_2 sample (SiO_2), and a nanostructured silicon electrode of the invention as described in Example 3 (as-deposited Si), and also provides an average of the silicon and SiO_2 spectra ($1/2(I_{Si} + I_{SiO2})$). Figure 5B provides an EELS spectrum showing the oxygen K-edge of the nanostructured silicon electrode described in Example 3.

6. A comparison of the spectra in Figure 5A indicates that the nanostructured silicon electrode has a SiO_2 component. The oxygen K-edge shown in Figure 5B also confirms the presence of SiO_2 in the nanostructured silicon electrode.

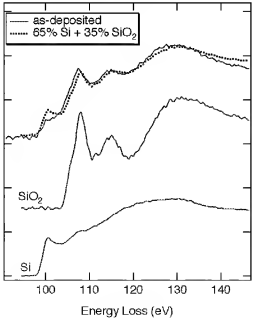
7. The electron energy loss spectra in Figures 5A and 5B may be analyzed to quantify the amount of SiO_2 present in the nanostructured silicon electrode. Two methods were used to determine the amount of SiO_2 in the nanostructured silicon electrode.

   The first method takes the integrated intensity of the background subtracted silicon $L_{2,3}$ edge and the oxygen *K* edge weighted by the scattering cross sections. This procedure gives a range from 50% - 67% by weight of SiO_2 from three different regions analyzed. The results from this method are very sensitive to the sample thickness and background subtraction.

   The second method relies on the near-edge structure from a single edge (silicon $L_{2,3}$). In this method the concentration of SiO_2 is determined by

qualitatively fitting the near-edge structure of the silicon $L_{2,3}$ edges from a plurality of different regions within the as-deposited nanostructured electrode. As shown in Figure 5A the near-edge structure from samples of pure Si and SiO_2 are quite different. A weighted sum of the spectra from Si and SiO_2 were qualitatively fit to the spectrum from the as-deposited nanostructured silicon electrode as shown in figure 5A. Supplemental Figure 1 (See, below) provides an overlay plot showing the results of fitting the weighted sum of the spectra from Si and SiO_2 to the EELS spectrum from the as-deposited nanostructured silicon electrode as shown in figure 5A. As shown in Supplemental Figure 1, the fit agrees very well with the observed EELS spectrum for the as deposited nanostructured electrode. The lowest SiO_2 concentration observed showed a good fit with 35% of the SiO_2 edge and 65% of the Si edge, suggesting the nanostructured silicon electrode consists of about 35% by weight SiO_2.

Supplemental Figure 1



In combination, these two analysis methods give us a range of between **35-67% by weight of SiO_2** for the as-deposited nanostructured electrode of Example 3.

8. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the above-identified U.S. Patent Application No. 10/660,382 or any patent issuing thereon.

Date: 06/22/2009

Jason Graetz